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Dislocation boundary formation and effect of high angle boundaries in nano copper crystals during in-situ TEM deformation

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A universal pattern has been established for the microstructural evolution with increasing strain in metals of medium to high stacking fault energy, such as copper. The structural scale covered in these studies spans from hundreds of micrometers to around a few micrometers [1,2] and there is a correlation between the deformation microstructure and the crystallographic orientation of the grains in which they evolve. The grain orientation affects the mechanical properties through its influence on the active slip systems, and therefore also the characteristics of the dislocations multiplying and gliding during plastic deformation. These dislocations are responsible for the formation and evolution of dislocation boundaries. Moreover, slip also leads to rotations of the crystallographic lattice, giving rise to texture evolution at the bulk scale. The recent interest in the behavior of sub-micron samples raises the important question of whether this coupling also extends to smaller length scales, as for example during the testing of submicron sized pillars. On even smaller samples, with sub-micrometer dimensions thin enough to allow transmission of electrons, dislocations have been imaged directly in the transmission electron microscope [3], including the recording of in-situ movies of gliding dislocations.

The dynamic quantitative in-situ mechanical testing in TEM allows the direct observation of dislocation activities and microstructural evolution with different imaging techniques, and of crystal rotation with diffraction techniques [4]. The imaging techniques include bright field imaging and dark field imaging. The diffraction techniques include convergent beam electron Kikuchi diffraction (CBEKD) for a specific point in the specimen and selected area electron diffraction (SAED) for an interested area. The direct observations of deformation processes further the understanding on the mechanical response of the different microstructural configurations in the nanoscale. This dynamic quantitative in-situ mechanical testing in the TEM was also favored by the recent development of the site-specific sampling techniques such as focused ion beam (FIB) milling technique especially in the case of specific microstructure pick-up.

Six nano copper pillars with square cross sections were cut from a small plate of recrystallized polycrystalline pure copper by FIB milling in a Zeiss 1540 dual beam microscope at 30 kV. This plate was sliced from a large slab, then mechanically ground to a thickness of 80 μm and glued to a module designed for in-situ uniaxial compression testing. The plastic deformation behavior of these pillars was characterized by in-situ compression in the TEM using a Hysitron PI-95 PicoIndenter. The ion-beam current was set at 1 nA for coarse milling and 5pA for fine milling. The exact dimensions of the pillars investigated were 545×220×214 nm^3, 485×229×213 nm^3, 600×231×254 nm^3, 608×210×167 nm^3, 600×174×174 nm^3 and 587×200×189 nm^3 for pillar 1, 2, 3, 4, 5 and 6, respectively. The crystallographic orientations of pillar 1, 2, 3 and 4 were identical, with the compression axis. Pillar 4 was bi-crystal with a 12° rotation around the compression axis and pillar 5 has four crystallites with boundary misorientation angles between neighboring crystallites of 28°, 52° and 40°, respectively. Compression tests were carried out in the displacement mode with the loading and unloading speeds of 2 nm/s. For pillar 1, 3, 4, 5 and 6, the normal loading-unloading route was applied while for pillar 2 the loading-holding-unloading route was applied. To record the dislocation activities and microstructural evolution, bright field imaging was used for pillar 1, 2, and 6, dark field imaging was used for pillar 3 and 5. CBEKD mode was used on the pillar 2 to record the in-situ variation of crystallographic orientation. These pillars were designed for different purposes: pillar 1 and 2 for observing the formation of dislocation boundaries, pillar 3 for identifying the dislocation activities, pillar 4 for the in-situ orientation variation, pillar 5 and 6 for investigating the effect of high angle boundaries on the mechanical responses. The in-situ observations were supplemented by pro- and post-test TEM analysis to determine the crystallographic orientation and Burgers vectors of active dislocations.

Low yield stress and large stress drops were observed in pillar 1, 2, 3 and 4, which was associated with a formation of low angle boundaries. Movies of deformation processes of pillar 1 and 2 show the dislocation generation, motion, escape of dislocations, and formation processes of dislocation boundaries. A video of dark field images of pillar 3 reveals the expected dislocation activities based on a Schmid factor analysis. In-situ variation of Kikuchi patterns of pillar 4 shows the crystal rotation during the deformation. These observations, combined with post-test TEM analysis to determine the crystallographic orientation and Burgers vectors of active dislocations (Fig. 1), show that the direction of crystallographic rotation during deformation is in agreement with slip taking place on a subset of the four slip systems, with the highest Schmid factors on the (111) and (-1-11) slip planes. However, the mechanical responses of pillar 5 and 6 are different with the previous four pillars, and only small flow stress drops were observed in pillar 5 and 6 and the strength has been improved by 2-3 times compared with pillar 1, 2, 3 and 4. Dark field video of pillar 5 shows the high angle boundary sliding assisted with the dislocation slip on the plane parallel to the boundary in the crystalline at the bottom end. Bright field video of pillar 6 demonstrates a more homogeneous deformation of the whole pillar from the beginning of the deformation.

The formation of a dislocation boundary in the nanoscale single copper crystal is discussed, and the significant effect of grain boundaries in the crystals is analyzed by combining the observations of microstructure, crystal rotation and strength as the plastic strain is increased.

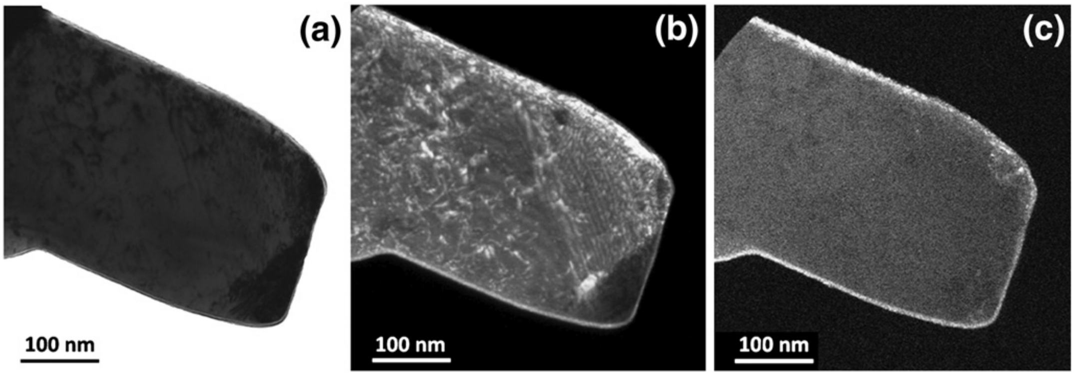

Figure 1. Bright field (a) and dark field TEM images of pillar 2 after deformation using $g_{(200)}$ (b) and $g_{(020)}$ (c) [5].

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